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Transport of Metals (Al, Fe) and Trace Elements (Cu, Mo, Ni, and Zn) through Intact Soil Cores Amended with Fresh or Composted Beef Cattle Manure for Nine Years

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ABSTRACT. Composting of feedlot cattle (Bos Taurus) manure may enhance metal and trace element accumulation and transport through the soil because these elements are concentrated in manure during composting. Little research has been conducted on comparing transport of metals (Al, Fe) and trace elements (Ni, Cu, Mo, Zn) through soil amended with composted manure (CM) versus fresh feedlot manure (FM) stockpiled for up to two months. Our objective was to determine if the transport of six selected chemicals (Al, Fe, Ni, Cu, Zn, Mo) was affected by the composting of cattle manure applied annually at 77 Mg ha^{-1} dry wt. for nine years to a clay loam soil. Intact soil cores were taken from a field experiment in the spring of 2007. Deionized water was applied to the soil cores in the laboratory under steady-state (4.9 cm d^{-1}) and unsaturated conditions. The chemical concentrations were measured in the effluent and breakthrough curves and cumulative mass loss curves obtained. Flow-weighted mean concentrations (FWMC) and mass loss of Al, Fe, Ni, Mo, and Cu, recovery of total applied Al, and maximum concentrations of Fe and Mo were significantly ($P \leq 0.05$) greater for CM compared to FM. Although greater chemical concentrations in amendments and soil for CM than FM may partially explain greater transport under CM, we believe that greater unsaturated hydraulic conductivity at 7 mBar for CM was a more important factor.

INTRODUCTION

Many feedlots in western Canada are interested in alternatives to the traditional method of land application of raw feedlot manure to cropland that is used for silage production. Some feedlots in southern Alberta are composting or stockpiling manure and are applying these amendments to cropland instead of fresh manure (Larney et al. 2006). However, the effect of long-term application of composted feedlot manure (CM) to cropland instead of fresh feedlot manure (FM) on accumulation, transport, and leaching of metals (Al, Fe) and trace elements (Ni, Cu, Mo, Zn), and the potential impact on soil and groundwater are unknown. Copper and Zn are added to feedlot cattle diets to minimize disease risk and improve feed efficiency (Larney et al. 2008; National Research Council 2000). Metals and trace elements in manure may also originate from soil ingestion or contamination during manure collection, processing, and storage (Bolan et al. 2004).

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National Canadian water quality guidelines exist for Al, Cu, Ni, and Zn with respect to community drinking water by humans, protection of aquatic life in surface water, irrigation water, livestock drinking water, and agricultural soils (CCME 2002). The only exceptions are Ni where there is no community drinking guideline for humans, and Al where there is no guideline for agricultural soils. Chemicals that can be translocated from soils through the human and animal food chain include Ni, Cu, Zn, and Mo (Page and Chang 1990).

Most researchers have generally reported that composting of cattle manure increases most total metal and trace element contents because of decomposition of organic matter (i.e., carbon mineralization) and a decrease in total mass of solid material in the final compost, but exceptions have also been reported (Fauci et al. 1999; Inbar et al. 1993; Larney et al. 2008). However, greater total concentrations of metals and trace elements in composted or fresh manure may not be a reliable indicator for predicting transport of these chemicals through soils by water (Hsu and Lo 2001). The chemical form or fraction, rather than total concentration, may be more important in determining leaching potential of different organic amendments (Hsu and Lo 2001; Petruzzelli et al. 1989). Hsu and Lo (2001) classified the leachability of metal fractions in decreasing order as water-soluble > exchangeable and organically-complexed > organically bound and solid particulate fractions > residual. In addition, water-soluble metals were not found to be related to total metal concentrations in fresh or composted swine manure (Hsu and Lo 2001).

Although most metals are immobile in managed agricultural soils, various factors that enhance mobility could result in more transport and leaching (McBride 1995). These factors include metal properties, soil binding sites, pH, concentration of complexing anions (organic and inorganic), and competing cations in soil solution (Tyler and McBride 1982). Aerobic composting increases formation of stable metal-humus complexes and generally leads to decreased solubility and mobility (Smith 2009). However, metal-organic complexes can become more soluble at a pH greater than 7 (McBride 1994), and this process is enhanced by dissolved organic matter (Ashworth and Alloway 2004).

We are unaware of any research that has compared transport of metals and trace elements through cropland soils that have been amended annually over the long-term with composted versus fresh feedlot manure. However, the accumulation and redistribution of total and EDTA (ethylenediaminetetraacetic acid)-extractable Cu and Zn through soils amended with increasing rates of fresh feedlot manure under dryland (30, 60, 90 Mg ha^{-1}) and irrigated (60, 120, 180 Mg ha^{-1}) conditions has been examined in the field after 11 years (Chang et al. 1991) and 25 years (Benke et al. 2008) of annual applications. These two studies reported that increasing manure application significantly increased EDTA-extractable Zn but not Cu, and there was no evidence of these trace elements being redistributed downward through the soil profile. Transport of nutrients and soluble salts through intact soil cores taken from a long-term field experiment comparing fresh versus composted feedlot manure has been studied (Miller et al. 2008, 2011, 2013); however, we are unaware of any lab column studies that have focused on metals and trace element transport from compost amended soils. The objective of our study was to determine if long-term (nine years) application of composted feedlot manure increased transport of selected metals and trace elements compared to fresh feedlot manure that was stockpiled for up to two months.

MATERIALS AND METHODS

The materials and methods for this leaching study have been previously described by Miller et al. (2011, 2012a) and only the main features relevant to this study will be described in this article. The field experiment was conducted on a clay loam Dark Brown Chernozemic soil (Typic Haploboroll) at Lethbridge, Alberta (Lat. 49° 38′ N; Long. 112° 48′ W). Long-term (1971–2000) annual precipitation at Lethbridge is 379 mm.

The treatments (four replicates) were initiated in 1998 and consisted of 56 field plots amended with three rates of fresh and composted feedlot manure containing straw or wood-chip bedding, inorganic N and P fertilizer, or an unamended control (CON). The organic amendments and

inorganic fertilizer were annually applied in the fall to irrigated silage barley. The fresh manure was stored for up to two months prior to land application. The composted manure was composted using the windrow composting method (Larney et al. 2003) using practices suggested by Rynk (1992). The windrows were turned about seven times over a 90-d period (active phase), and then entered a curing phase (no turning) for a further 90 to 120 d until compost temperatures reached ambient air temperatures.

For our laboratory column study, we focused on 12 field plots consisting of two organic amendment treatments and the unamended control (CON). The two organic amendment treatments were 77 Mg ha^{-1} rate of fresh manure (FM) and composted feedlot manure (CM) with straw bedding. Intact soil cores were taken using a truck-mounted drill from four replicates of each treatment and the control, for a total of 12 soil cores. The soil cores used were acrylic tubes 30.3 cm in length, 8.9 cm in diameter, and 0.4 cm wall thickness. The soil cores taken were then sealed with plastic wrap and stored at 0.5°C, as microbial activity in the soil at this temperature is minimal (Havlin et al. 1999). The soil cores were stored for a period of between two to four months before the transport experiment was conducted on each core.

The leaching apparatus consisted of a syringe pump, soil column (with undisturbed soil core) and flow cell, fraction collector, vacuum chamber, vacuum regulator, and elbow pencil tensiometers (Soil Measurement Systems®, Tucson, AZ, USA). All experiments were done under unsaturated and steady-state flow conditions using an inflow rate of 4.9 cm d^{-1} (deionized water) and a water tension of approximately 7 mBar. Leachate was collected in the fraction collector and stored at 4°C until analyzed within one to six weeks. Since the same volume increments of effluent and total volume were collected for all soil cores, mean concentrations of chemicals in effluent are equivalent to flow-weighted mean concentrations (FWMC), which are used to normalize concentrations for different flow rates.

Water-soluble metals (Al, Fe) and trace elements (Cu, Mo, Ni, Zn) in amendments and soil were extracted using a 1:5 (20 g manure:100 mL water) ratio and 1:2 (30 g soil:60 mL water) ratio, respectively. Water-soluble concentrations of metals and trace elements in water extracts and leaching effluent were determined by inductively coupled plasma-optical emission spectrometry (ICP-OES) with axially viewed plasma (Spectro Analytical Instruments, Kleve, Germany).

Statistical analyses

A MIXED model analysis (Littell et al. 1998) in SAS (SAS Institute 2005) was used to analyze the maximum concentrations, FWMC, mass loss, percentage recovery of applied chemicals, and percentage recovery of chemicals in the soil. Treatment was considered as a fixed effect and soil core replicates as random effects in the model. Treatment effects were considered significant at the $P \leq 0.05$ level, and means comparisons were conducted using least squares means (LSM). When required, the MIXED model analysis was conducted on log-transformed values.

RESULTS

The concentration of Al, Fe, Cu, and Zn were 6 to 17% greater in the CM compared to FM amendment before they were added to the soil (table 1). In contrast, Ni and Mo concentrations were 7 to 10% greater in the FM than the CM amendment. Concentrations of the six metals and trace elements were 16 to 39% greater for soil amended with CM compared to FM just prior to this lab experiment (table 1). The exception was Mo where it was 18% greater for FM than CM. The pH of the amendments was generally similar for CM and FM, and soil pH values were also similar for the three treatments (table 1).

Maximum concentrations of Fe and Mo in effluent were significantly greater by 1.6- to 2.9-fold for CM compared to FM (table 2, figure 1). However, maximum concentrations of Al, Ni, Cu, and Zn were similar for CM and FM (figure 1). Maximum concentrations in the amended soils were significantly greater by 3.5- to 101-fold compared to the unamended CON and significant differences were observed for all

TABLE 1. Concentrations of Al, Fe, Ni, Cu, Zn, and Mo of fresh (FM) and composted (CM) feedlot manure, means from 1998 through 2006 and soil (0–30 cm) from three treatments prior (June 4, 2007) to leaching experiments

Treatment*	pH	Al (mg kg^{-1})	Fe (mg kg^{-1})	Ni (mg kg^{-1})	Cu (mg kg^{-1})	Zn (mg kg^{-1})	Mo (mg kg^{-1})
Amendment							
FM	8.3 ± 0.1	6462 ± 436	8984 ± 540	68.4 ± 9.7	42.4 ± 2.3	176.6 ± 13.4	7.2 ± 0.82
CM	8.1 ± 0.1	7128 ± 438	$10,481 \pm 691$	63.6 ± 13.7	45.1 ± 2.2	192.1 ± 14.4	6.5 ± 1.1
Soil							
CON	7.9 ± 0.04	3.1 ± 0.63	4.0 ± 0.71	0.06 ± 0.0008	0.10 ± 0.003	0.016 ± 0.002	0.036 ± 0.001
FM	7.9 ± 0.03	157 ± 48	96 ± 28	0.31 ± 0.02	0.58 ± 0.018	0.50 ± 0.086	0.22 ± 0.018
CM	8.0 ± 0.04	192 ± 44	118 ± 30	0.43 ± 0.02	0.67 ± 0.06	0.62 ± 0.06	0.18 ± 0.04

*CON: unamended control; FM: fresh manure; CM: composted manure.

TABLE 2. Manure type effects on flow-weighed mean concentration (FWMC), maximum concentrations, cumulative mass loss, and recovery of applied trace elements in effluent from soil cores as a percentage of total applied or total in soil

Trace element	Treatment* CM	FM	Control	Prob > F†
Max. conc.				
Al (mg L^{-1})	4.04 ± 0.82 a	2.24 ± 0.70 a	0.04 ± 0.0 b	0.0002
Fe (mg L^{-1})	4.89 ± 1.18 a	1.68 ± 0.57 b	0.11 ± 0.0 b	0.0046
Ni (μg L^{-1})	57.7 ± 5.2 a	130 ± 62.5 a	13.4 ± 6.2 a	0.1266
Cu (μg L^{-1})	107 ± 12.8 a	74.3 ± 10.9 a	17.9 ± 5.7 b	0.0005
Zn (μg L^{-1})	53.5 ± 5.6 a	94.6 ± 54.7 a	15.3 ± 4.0 a	0.2631
Mo (μg L^{-1})	116 ± 17.9 a	74.5 ± 6.6 b	9.5 ± 0.0 c	0.0003
FWMC				
Al (mg L^{-1})	1.22 ± 0.094 a	0.35 ± 0.043 b‡	0.038 ± 0.0 c	<0.0001
Fe (mg L^{-1})	1.60 ± 0.09 a	0.67 ± 0.04 b	0.11 ± 0.0 c	<0.0001
Ni (μg L^{-1})	38.3 ± 0.9 a	31.4 ± 1.3 b	6.7 ± 0.2 c	<0.0001
Cu (μg L^{-1})	71.7 ± 2.0 a	43.8 ± 1.4 b	8.8 ± 0.3 c	<0.0001
Zn (μg L^{-1})	29.0 ± 0.9 a	28.4 ± 3.0 a	7.6 ± 0.2 b	<0.0001
Mo (μg L^{-1})	61.4 ± 2.6 a	39.3 ± 1.6 b	9.5 ± 0.0 c	<0.0001
Mass loss				
Al (kg ha^{-1})	7.8 ± 2.3 a	2.4 ± 0.7 b	0.2 ± 0.0 b	0.0010
Fe (kg ha^{-1})	9.7 ± 2.0 a	3.6 ± 1.0 b	0.5 ± 0.0 b	0.0022
Ni (g ha^{-1})	192 ± 12.1 a	142 ± 15.1 b	30.6 ± 2.6 c	<0.0001
Cu (g ha^{-1})	346 ± 56.9 a	202 ± 26.9 b	40.1 ± 7.8 c	0.0008
Zn (g ha^{-1})	133 ± 22.4 a	91.9 ± 36.0 a	36.9 ± 3.4 a	0.0624
Mo (g ha^{-1})	344 ± 55.5 a	223 ± 32.3 b	46.4 ± 0.0 c	0.0010
Recovery of total applied (%)				
Al	0.18 ± 0.05 a	0.03 ± 0.03 b	—	0.0321
Fe	0.13 ± 0.03 a	0.08 ± 0.03 a	—	0.2070
Ni	0.38 ± 0.03 a	0.33 ± 0.03 a	—	0.2070
Cu	0.28 ± 0.05 a	0.15 ± 0.03 a	—	0.0667
Zn	0.43 ± 0.08 a	0.33 ± 0.13 a	—	0.5334
Mo	7.7 ± 1.2 a	4.5 ± 0.7 a		0.0618
Recovery of total in soil (%)				
Al	1.1 ± 0.2 a	0.6 ± 0.2 a	—	0.1738
Fe	2.5 ± 0.5 a	1.8 ± 0.9 a	—	0.5528
Ni	11.8 ± 0.7 a	13.4 ± 2.3 a	—	0.5401
Cu	13.8 ± 2.9 a	9.9 ± 1.5 a	—	0.2697
Zn	5.5 ± 0.7 a	5.5 ± 2.3 a	—	0.9920
Mo	60.9 ± 18.7 a	28.5 ± 2.7 a	—	0.1376

*FM: fresh manure; CM: composted manure; Control: unamended control.
†Prob > F, probability greater than F statistic.
‡Mean ± standard error. FWMC: Al, Fe, and Mo = log transformed; others = arithmetic.
Maximum concentrations: Al = log transformed; others = arithmetic. Loads and recoveries: all untransformed.

chemicals except for Ni and Zn. The FWMC of Al, Fe, Ni, Cu, and Mo were significantly greater by 1.2- to 3.5-fold for CM compared to FM, and FWMC were similar for Zn (table 2). The FWMC of the chemicals were significantly greater by 3.7- to 32-fold for the amended soils compared to unamended CON.

The mass loss of all six chemicals except for Zn was significantly greater by 1.4- to 3.3-fold for CM compared to FM (table 2, figure 2). The mass loss was significantly greater by 2.5- to 39-fold for the amended soils compared to unamended CON. The exceptions were Al, Fe, and Zn, where mass loss was similar for FM and the CON. The recovery of Al as a percentage of total Al applied was significantly greater for CM compared to FM (table 2). In contrast, manure treatment had no significant influence on

FIGURE 1. Breakthrough curves of A, Al; B, Fe; C, Ni; D, Cu; E, Zn; and F, Mo under fresh manure (FM), composted manure (CM), and the unamended control (CON).

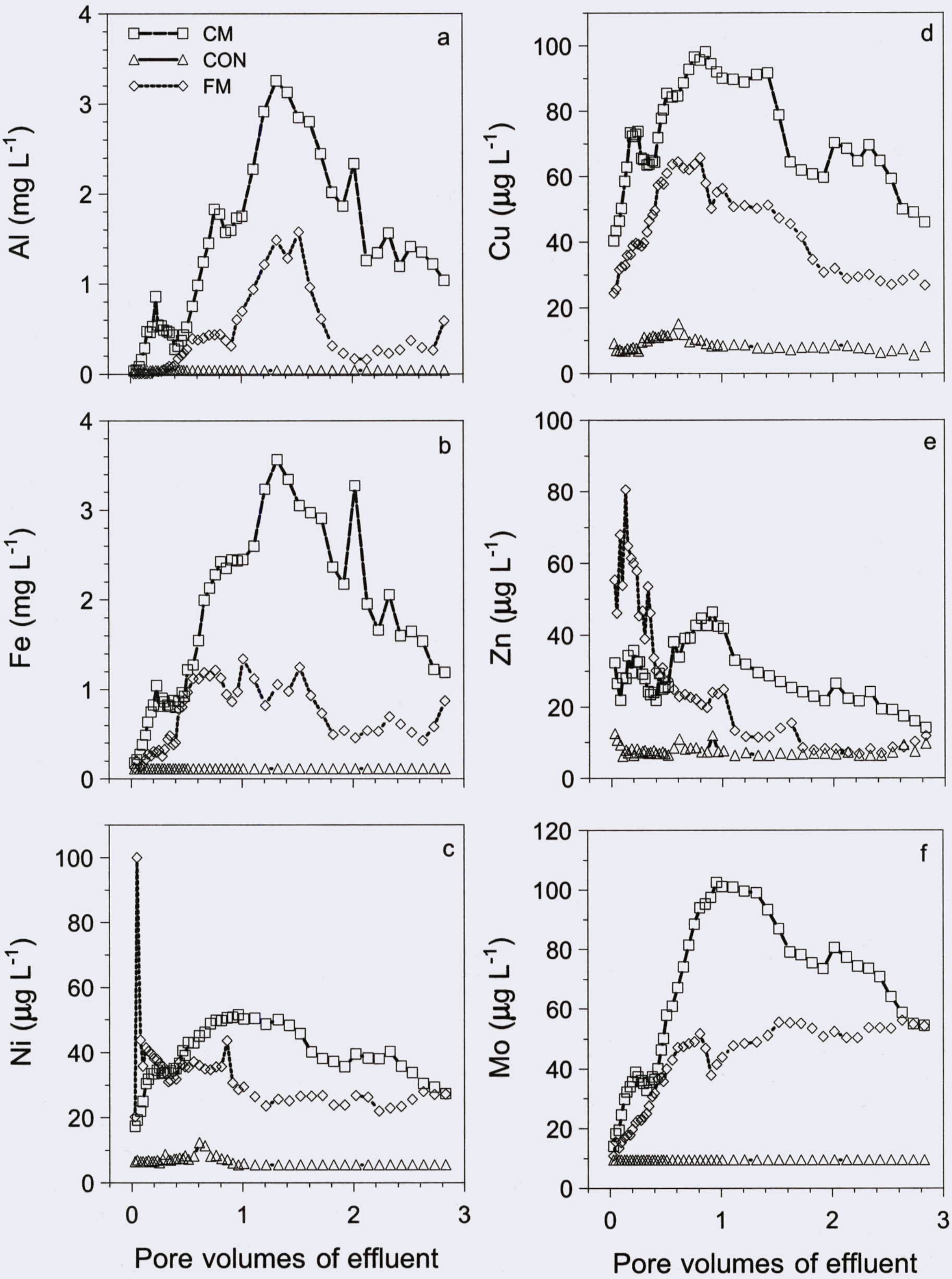
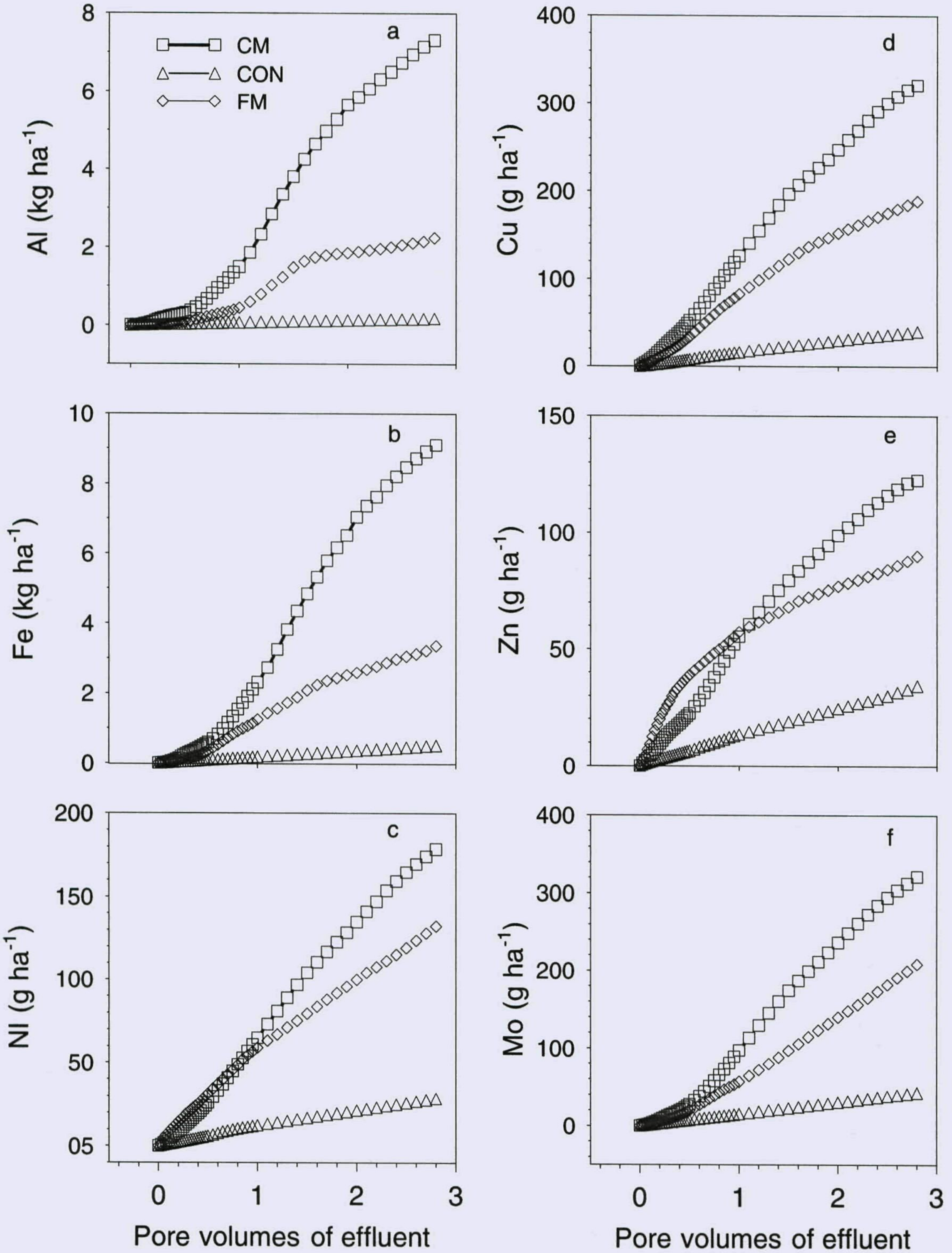


FIGURE 2. Cumulative mass of A, Al; B, Fe; C, Ni; D, Cu; E, Zn; and F, Mo in leachate under fresh manure (FM), composted manure (CM), and the namended control (CON).



the other five chemicals. In addition, the recovery of the six chemicals as a percentage of total concentrations in the soil was similar for CM and FM.

The maximum concentrations and FWMC of Al, Cu, Ni, Mo, and Zn generally exceeded the water quality guidelines for drinking water and aquatic life considerably more for CM compared to FM and the unamended CON (table 3). For the three elements (Al, Cu, Zn) where community drinking water guidelines existed, Al exceeded the guideline and Cu and Zn did not exceed the guideline (table 3). Aluminum also exceeded the aquatic life guideline by the largest factor, followed by Cu, Ni, Zn, and Mo. Molybdenum exceeded the irrigation guideline by the largest factor, whereas the other five chemicals did not exceed this guideline. None of the five chemicals exceeded the livestock guideline.

DISCUSSION

Statistical analysis of our results indicated that we could accept our hypothesis of greater chemical transport for CM compared to FM for FWMC and mass loss of Al, Fe, Ni, Cu, and Mo, recovery of total Al applied, and maximum concentrations of Fe and Mo. These findings also suggested that metal and trace element transport for these two treatments was dependent on how the effluent transport was determined: concentration, mass loss, the recovery of applied chemical, or recovery of chemical in soil.

Greater transport of all metals and trace elements (except Zn) under CM than FM may be partially related to slightly greater concentrations of certain elements in the amendments or soil. For example, Al, Fe, and Cu were 6- to 39-fold greater in CM compared to FM amendments or soil. In contrast, certain trace elements (Ni, Mo) were greater in amendments or soil for FM compared to CM, suggesting that the amendments or soil could not explain greater transport under CM. For example, Ni and Mo concentrations were 7 to 10% greater in FM compared to CM amendments; and Mo was 18% greater in soil amended with FM compared to CM.

Our results of generally greater transport of most metals under CM compared to FM did not support previous findings that aerobic composting increases formation of stable metal-humus complexes and generally leads to decreased solubility and mobility (Smith 2009). This may have been due to the alkaline pH of our amendments (8.1–8.3) and amended soil (7.9–8.0). In addition, metal-organic complexes can become more soluble at a pH greater than 7 (McBride 1994), and this process is enhanced by dissolved organic matter (Ashworth and Alloway 2004). The water-soluble organic carbon concentration in our soil was similar (1.1 $g\ kg^{-1}$) under CM and FM (Miller et al. 2012), suggesting no manure type effect on dissolved organic matter.

We believe that the greater transport of most trace elements in soil amended with CM compared to FM was likely due to the greater water flow through CM soil. Miller et al. (2013) reported that unsaturated hydraulic conductivity at 7 mBar was significantly greater for CM compared to FM by 40%. In addition, significantly greater transport under CM than FM for NO_3, Cl, total reactive P, soluble salts (Na, K), and total S in these same soil cores (Miller et al. 2011, 2013) was additional evidence that differences in water flow rather than chemical differences in amendments or soil were causing greater metal and trace element transport under CM.

Certain maximum concentrations and FWMC of trace elements exceeded the federal water quality guidelines (CCME 2002) for community drinking water by humans, protection of aquatic life, and irrigation water (table 3). In addition, when water quality guidelines were exceeded, the level of the exceedance was generally greater for CM compared to FM.

Maximum concentrations and FWMC of Al (but not Cu or Zn) in effluent that reaches the groundwater or surface waters may be a potential problem for community drinking water by humans. The following metals or trace elements may be a potential problem for aquatic life if this effluent discharges into surface waters without further dilution. These include maximum concentrations and FWMC of Al and Cu in effluent from amended and unamended soils, maximum concentrations and FWMC of Ni in amended soils, maximum Mo concentrations in

TABLE 3. Magnitude of exceedances of maximum concentrations and flow-weighted mean concentrations (FWMCs) in relation to federal water quality guidelines (WQG) for community water (drinking by humans), protection of aquatic life, irrigation of crops, and livestock consumption (shaded values > 1.0 indicate that water quality guideline was exceeded; values ≤ 1.0 indicate that the guideline was not exceeded)

	Magnitude of exceedance*			
	Community	Aquatic life	Irrigation	Livestock
Al-max.				
CM	40.4	808	0.8	0.8
FM	22.4	448	0.4	0.4
CON	0.4	8.0	0.0	0.0
Al-FWMC				
CM	12.2	244	0.2	0.2
FM	3.5	70	0.1	0.1
CON	0.4	7.6	0.0	0.0
Al-WQG (mg L^{-1})	0.1	0.005–0.1	5	5
Cu-max				
CM	0.1	53.5	0.5	0.2
FM	0.1	37.2	0.4	0.1
CON	0.0	9.0	0.1	0.0
Cu-FWMC				
CM	0.1	35.9	0.4	0.1
FM	0.0	21.9	0.2	0.1
CON	0.0	4.4	0.0	0.0
Cu-WQG (ug L^{-1})	1000	2–4	200–1000	500–5000
Ni-max.				
CM	—	2.3	0.3	0.1
FM	—	5.2	0.7	0.1
CON	—	0.5	0.1	0.0
Ni-FWMC				
CM	—	1.5	0.2	0.0
FM	—	1.3	0.2	0.0
CON	—	0.3	0.0	0.0
Ni-WQG (ug L^{-1})	—	25–150	200	1000
Mo-max.				
CM	—	1.6	11.6	0.2
FM	—	1.0	7.5	0.1
CON	—	0.1	1.0	0.0
Mo-FWMC				
CM	—	0.8	6.1	0.1
FM	—	0.5	3.9	0.1
CON	—	0.1	1.0	0.0
Mo-WQG (ug L^{-1})	—	73	10–50	500
Zn-max.				
CM	0.0	4.4	0.1	0.0
FM	0.0	3.1	0.1	0.0
CON	0.0	1.2	0.0	0.0
Zn-FWMC				
CM	0.0	1.0	0.0	0.0
FM	0.0	0.9	0.0	0.0
CON	0.0	0.3	0.0	0.0
Zn-WQG (ug L^{-1})	5000	30	1000–5000	50,000

*Magnitude of exceedance is ratio of maximum concentration or FWMC (table 2) divided by the pertinent CCME (2002) water quality guideline. The minimum value of WQG was used when a range of values were given.

CM soil, and maximum Zn concentrations in amended and unamended soil. Maximum concentrations and FWMC of Mo in amended soils may be a potential problem for irrigation water if this effluent leaches into the groundwater. In contrast, no concentrations of trace elements exceeded any of the guidelines for livestock water consumption.

Concentrations exceeding various water quality guidelines suggested a potential for groundwater and surface water pollution by certain trace elements. However, this assumes that these elements at the 30 cm depth can actually reach the groundwater or flow laterally through soils into surface waters and is not diluted. There is a potential for leaching of metals and trace elements to the shallow water table by macropore flow because these pores, ≥ 1 mm in diameter, were visually observed throughout the root zone of this experiment during the time of soil core sampling, and the water table is shallow (1 to 2 m) under irrigation adjacent to this site (C. Chang, 2010, personal communication). However, breakthrough curves for all six trace elements were not asymmetrical and maximum concentrations generally did not occur prior to one volume as was found for nutrients, soluble salts, and total S (Miller et al. 2011, 2013). This suggested a lower potential for preferential flow of metals and trace elements in macropores compared to these other more mobile chemicals. Most positively charged metals are generally immobile in agricultural soils (McBride 1995). However, preferential flow and organic-facilitated transport can accelerate metal leaching through soils (Camobreco et al. 1996).

CONCLUSIONS

We can accept our hypothesis of greater chemical transport for CM compared to FM for FWMCs and mass loss of Al, Fe, Ni, Cu, and Mo, recovery of total Al applied, and maximum concentrations of Fe and Mo. Differences in concentrations of metals and trace elements in the amendments and soil may partially explain our findings. However, we believe that the most likely explanation for greater transport of certain metals and trace elements was the significantly greater unsaturated hydraulic conductivity at 7 mBar through soils amended with CM compared to FM. Maximum concentrations and FWMC of certain metals and trace elements in effluent may exceed water quality guidelines for community drinking water (Al), aquatic life (Al, Cu, Ni, Mo, Zn), and irrigation water (Mo). This assumes that this effluent actually reaches the groundwater or surface water and is not diluted. Breakthrough curves suggested that the potential for preferential flow of metals and trace elements in macropores was generally considerably lower for these chemicals compared to nutrients, soluble salts, and total S that was reported previously.

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